STK1094
Analytical Chemistry I

Laboratory Manual

lty of Resource Science & Technology
Universiti Malaysia Sarawak

Contents

Example of cover page

FACULTY OF RESOURCE SCIENCE AND TECHNOLOGY
DEPARTMENT OF CHEMISTRY

STK 1094 –Analytical Chemistry I

EXPERIMENT NO:

TITLE OF EXPERIMENT:

DATE OF EXPERIMENT	:	
GROUP MEMBERS & MATRIC NUMBERS	:	
LAB FACILITATOR	:	
REPORT DUE DATE	:	

Laboratory Outline

1. The lab report should be **organized** according to the following sequence:
    a) Introduction
    b) Objectives
    c) Procedures and material/apparatus
    d) Results
    e) Calculations, if any
    f) Discussion
    g) Conclusion
    h) Post-lab questions
    i) References

2. **Unorganized** lab report will be **penalized**.
3. The content of each section in a lab report is described in Table 1.
4. Only **one** lab report needs to be submitted **per group.**
5. Lab report is to be **submitted** at the **beginning** of **next lab session**.
6. **Late** submission will be **penalized**.
7. **Plagiarism** is strictly **prohibited**.

Table 1: Description on the content of a lab report

Section	Description
Introduction	• Brief discussion on the background of the technique(s) used in the experiment. • The theory behind any calculation involved should be presented in the introduction as well. • Any referred information must be cited.
Objectives	Brief description on the objectives of the outlined experiment
Experimental set up and procedure	• Consists of the list of chemicals and apparatus used. • All steps performed in the experimental procedure should be listed in the order that they were performed, in exactly the manner in which you performed them. • The procedures must be written in passive sentences.
Results	• Should list all data obtained, in raw form, with information provided as to how the data was obtained, as well as the accuracy of all measurements. • Record the numbers/measurements you collect (Quantitative data) • Record other pertinent observations (Qualitative data) • If possible use tables. • Include graphs if appropriate. • Include ALL, even those that will be rejected later.
Calculation (if any)	• Calculation should be included after the results shown. • Include formulas, units, use significant digits. • Show complete calculations for all results.

Discussion	• The data should be discussed and evaluated, both positively and negatively. • Do not try to manipulate data to fit the results you think you should obtain. • Evaluate the data fairly, even if the data seem to contradict with theory you may have been expecting the data to follow. • A discussion of possible sources of error should be included in this section. • Calculate your percent error, if applicable. • If any data was rejected, explain why here. • Any referred information must be cited. • Include chemical reactions involved (if any).
Conclusion	• Concise, direct statement of what you learned. • If possible, use a single sentence.
Post-lab questions	• Answer all the post-lab questions provided after each experiment.
References	• All materials that are used in writing the laboratory report should be listed (at least two). • Journal articles are referenced by listing the authors (last name first), the year of publication, the title of the article, the title of the journal, the volume number and the page number. For example: *Kuivinenm J., Johnsson, H. (1999) Determination of Trihalomethanes and some Chlorinated solvents in Drinking Water. Water Research, 33 (5). 1201-1208.* • Books are referenced by listing the authors (last name first), year of publication, the title of the book, the edition, the publisher, city of publication and the page number. For example: *John, M.M., Johnston, D.O., Nettervile, J.T., Wood, J.L., Joesten, M.D. 1991. Laboratory Manual to Accompany World of Chemistry. Sounders College Publishing, New York.*

Laboratory Safety

Safety in the laboratory is a subject of the utmost importance. All chemicals are harmful to some degree, therefore it is imperative to learn the safety rules and follow them strictly at all times. You will be expelled from the laboratory for failing to comply with these regulations. These rules are referred by many laboratories as "the usual safety procedures".

General

1. Wear shoes at all times when you are in the laboratory.
2. Wear lab coat at all times when you are in the laboratory
3. Report any spill or accident immediately to your instructor.
4. Know the location and operation of safety equipment in the laboratory from the first meeting of the laboratory session.
5. Drinking, eating and smoking are absolutely forbidden in the laboratory.
6. Never work alone in the laboratory.
7. Dispose the chemicals properly, in the container provided, and according to the instructions given by the laboratory instructor. Do not simply pour chemical wastes down the sink.
8. Keep your laboratory space clean.

Safety glasses

1. Safety glasses should be worn at all times while in the laboratory.
2. Contact lenses should never be worn in the laboratory because they cannot be removed rapidly if reagents accidentally splash in the eye.

Chemicals

1. Handle all chemicals according to any specific directions indicated on the container, or those given to you by your instructor.
2. Avoid contact with skin and clothing.
3. Wipe up spills immediately, especially near the balances and reagent shelf.
4. Replace caps on containers immediately after use.
5. Avoid the inhalation of organic vapours, particularly aromatic solvents and chlorinated solvents.

Disposals of chemicals

1. Dispose of chemicals as directed in each experiment.
2. Water-soluble substances can be flushed down the drain with large quantity of water.
3. Water insoluble solids and liquid should be placed in the waste container provided.
4. Chromium ion in the +6 oxidation state should be reduced to the +3 state with a mild reducing agent before disposal.

Apparatus and techniques

The following is a summary of the basic analytical laboratory techniques and equipment you will use for this semester. Proper techniques are essential as acceptable error in a quantitative chemical analysis is seldom greater than 0.1 %. There are a number of "hard and fast" rules presented that must be followed to minimize any hazards to yourself, your lab-coworkers, and the lab equipment. **Read this section at the beginning of the semester and refer to any of this material as often as necessary.**

I. "Clean" and "Clean and Dry" glassware

You will notice throughout the semester that you are asked to use "Clean" glassware at times and "Clean and Dry" glassware at other times. "Clean" glassware may be wet with your solvent (usually distilled water), and most of the times it is not worth the effort to dry the piece of glassware.

In general, if you want to maintain the concentration of the solution being transferred, you will want the final container to be "Clean and Dry". However, if you are only concerned about the amount of the compound being transferred, the final container need only be "Clean".

"Clean" glassware means that all compounds and materials have been washed out. The final washing should be with your solvent. In analytical chemistry, the solvent is defined as the liquid or solution that you would use to dilute the solution in question, usually distilled water.

II. Desiccators and Handling Dried Compounds

When using primary standards (compounds that are presumed to be 100% pure) in analyses, it is essential that there are no crystal waters present so that the mass of the primary standard measured on the balance is equal to the actual mass. Typically, compounds are dried by placing them in an oven at 105 to 120 °C. After several hours, all (or most) crystal waters have been driven off. However hot compounds cannot be accurately weighed (***all items weighed on a balance must be at room temperature***), so there must be a way to cool a dried compound without re-exposing it to water vapor in the atmosphere.

Desiccators are containers designed to prevent the re-hydration of solids. The bottom half of the desiccator is filled with an anhydrous salt, such as calcium chloride. The dried compound and its container sit in the top half, which is separated from the bottom half by a grid or screen. The desiccator lid can be sealed with vacuum grease to prevent water vapor from seeping inside.

Always cool a dried compound in a desiccator before weighing. A dried compound can be kept in the desiccator if that compound has to be available throughout the lab period.

III. Electronic and Analytical Balances

Electronic balances are quite simple to use. As you probably know, the "tare" button resets the mass reading to zero, and there is usually another button (sometimes labeled "cal" for calibrate) to set the mass units. You will always want the mass units in grams. Because electronic balances are fragile, you need to observe the following guidelines.

1. Always clean the balance after using it -- use a soft brush or Kimwipe to remove any extraneous material from the balance pan.

2. All items and compounds placed on the balance must be at room temperature -- this can come into play when weighing dried compounds. Cool dried compounds in a desiccator before weighing them on the balance.

3. If you are making repeated weighing in the same container, it is recommended that you always tare the empty balance and record the mass of the empty container. Then, record the mass of the container with sample, and calculate the mass of the sample by difference.

4. If you are instructed to "accurately weigh" something, use a balance with 4 decimal places. This is referred to as analytical balance. The maximum capacity of an analytical balance is usually small (60 or 180 g), therefore use only weighing boats or weighing paper as containers on the balance. On the electronic balance with 3 decimal places, you can often use small beakers or flasks as containers.

IV. Volumetric Flasks and Quantitative Transfers

Volumetric flasks are calibrated to contain an exact volume of solution when the solution level is exactly at the mark on the neck of the flask (the bottom of the meniscus should lie exactly at this mark). Note the following rules in handling volumetric flasks.

1. ***To clean volumetric flasks*** -- Each washing should have a volume that is about 10 to 20% of the capacity of the vol. flask. Typically, you should wash the flask 3 times with dilute acid (e.g., 1 to 6 M HCl), 3 times with distilled water, and 3 times with your solvent (if it is not distilled water). You can skip the acid washings if you have no solid residue in the flask.

2. ***Never heat a volumetric flask*** -- heating causes the glass to expand, changing the volume it contains.

3. ***NEVER place a solid directly into a volumetric flask*** -- What do you do if you fill the flask to the mark and the solid won't dissolve? Dissolve the solid in another container and quantitatively transfer the solution to the volumetric flask. For example, if you want to make a 100-mL solution of NaCl, dissolve the NaCl in a beaker with 75 to 90 mL of water and then transfer this to the volumetric flask.

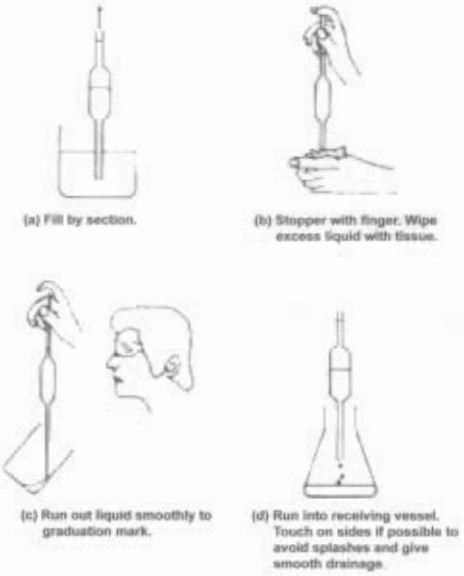
4. ***Never shake a volumetric flask*** -- when mixing a solution in a vol. flask, gently invert the flask 8 to 10 times.

5. ***Quantitative transfers*** are vital to accurate analyses. In simple terms, quantitatively transferring something means washing the original container and all glassware involved in the transfer with solvent and adding those washings to the final container, usually a volumetric flask. Here are some guidelines to transferring solutions and solids to volumetric flask.

Pour the solution through a funnel into the volumetric flask. Wash the original container with a small amount of solvent and pour the washing through the funnel into the volumetric flask. Repeat the washing 2 or 3 times if possible. Dilute the solution in the volumetric flask to the mark.

V. Volumetric Pipettes

Volumetric pipettes are calibrated to deliver an exact volume of liquid or solution. Volumetric pipettes have only one calibration mark. You may have seen graduated pipettes that have calibration marks throughout the length of the pipette, but **these are far less accurate than volumetric pipettes.** To fill a pipette, simply draw in liquid to the mark. Usually it is easiest to initially overshoot the mark and then let the liquid drain from the pipette until the bottom of the meniscus lies exactly on the calibration mark.



Note the following rules in handling pipettes.

1. ***Never pipette with your mouth!*** Always use a rubber bulb, regardless of what you are taught in biology classes.

2. ***Always clean a pipette before its initial use*** -- For each washing, draw liquid into the pipette so that the bulb is 1/4 to 1/2 full (this can be less for large pipettes). Carefully swirl the liquid throughout the inside of the pipette (don't let liquid pour out the top!), and let the liquid drain from the pipette. Wash the pipette 3 times with distilled water and 3 times with the solution you are going to transfer (not just the solvent). If you are using the same pipette for different solutions, you need to repeat the washing procedure every time you switch solutions.

3. ***Never force liquid out of a pipette*** -- always let the liquid drain of its own accord. The calibration mark takes into account any liquid that is retained in the tip of the pipette. When the liquid has stopped draining, touch the tip of the pipette against the side of the container to release any hanging drops.

4. At the end of a lab period, always wash used pipettes 3 times with distilled water.

VI. Burettes

Burettes allow you to accurately deliver volumes of liquid that cannot be measured by volumetric pipettes or micropipettors. The proper use of burettes is essential to accurate titration analyses. In several cases, an analyte can be determined more accurately using a titrimetric method rather than an instrumental method -- it's just that the convenience of the instrumental use is sometimes the deciding factor in choosing an analytical method. Note the following rules and guidelines in using burettes.

1. ***To clean a burette*** -- fill the burette with distilled water and drain a large portion of it to see if any water adheres to the inside walls of the burette. If so, clean the burette with a few milliliters of soap solution and a burette brush, and wash the burette with three 5-mL portions of tap water. When washing the inside of a burette, pour about 5 mL of liquid into the burette with the stopcock closed. Carefully swirl the liquid for a few seconds so that it comes in contact with the entire inside surface area of the burette, and pour the liquid out the top. If no water adheres to the inside walls of the burette, proceed to wash it 3 times with distilled water and 3 times with the solution with which you are going to titrate your sample.

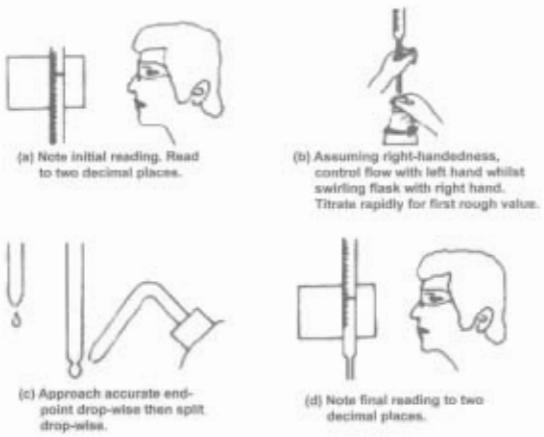
2. ***Getting rid of air bubbles*** -- fill the clean burette with your solution. There will be air bubbles inside the stopcock, and you **must** remove these (you can't measure the volume of an air bubble in a burette, so if an air bubble pops out in the middle of your titration, you're sunk). It's usually easiest to force bubbles out the stopcock, so simply open the stopcock and let the solution drain until the air bubbles are removed. If this isn't working, you can try **gently** tapping the base of the burette while the solution is draining. This may force the air bubbles to rise through the solution.

3. ***Always record volume levels to the nearest 0.01 mL*** -- Although the calibration marks are only at every 0.1 mL, you can always estimate the extra decimal place.

For Example:

The reading in this picture is between 8.2 and 8.3 mL. The second decimal place would be estimated to be about 0.07 mL, giving a reading of **8.27 mL**.

To obtain readings, it helps to hold a white card behind the burette. Note reading at a particular part of meniscus and always measure at this part.



4. ***NEVER record an initial volume level of 0.00 mL*** -- since there are no calibration marks above 0.00 mL, you have no upper reference with which to base any possible error in your reading.

5. Using your wash bottle, you can add "half-drops" to your titration flask (see above figure). Run a drop of solution part-way out of your burette tip. Squirt distilled water on this half-drop and into your titration flask. This procedure is perfectly legitimate because you aren't worried about the concentration of anything in your titration flask.

6.At the end of an experiment, always wash your burette 3 times with distilled water.

EXPERIMENT 1: ACID-BASE TITRATIONS

INTRODUCTION

The technique of titration finds many applications, but is especially useful in the analysis of acidic and basic substances. Titration involves measuring the exact volume of a solution of known concentration that is required to react with a measured volume of a solution of unknown concentration, or with a weighed sample of unknown solid. A solution of accurately known concentration is called a standard solution.

In many cases it is possible to prepare a standard solution by accurate weighing of the solute, followed by precise dilution to an exactly known volume in a volumetric flask. One of the most common standard solutions used in acid-base titration analysis, however, cannot be prepared in this manner.

Solutions of sodium hydroxide are commonly used in titration analysis of samples containing an acidic solute. Although sodium hydroxide is a solid, it is not possible to prepare standard sodium hydroxide solutions by mass. Solid sodium hydroxide is usually of questionable purity. Sodium hydroxide reacts with carbon dioxide from the atmosphere and is also capable of reacting with the glass of the container in which it is provided. For these reasons, sodium hydroxide solutions are generally prepared to be approximately a given concentration. They are then standardized by titration of a weighed sample of a primary standard acidic substance. By measuring how many mL of the approximately prepared sodium hydroxide are necessary to react completely with a weighed sample of a known primary standard acidic substance, the concentration of the sodium hydroxide solution can be calculated. Once prepared, however, the concentration of a sodium hydroxide will change with time (for the same reasons stated above). As a consequence, sodium hydroxide solutions must be used relatively quickly.

In titration analysis, there must be some means of knowing when enough titrant has been added to react exactly and completely with the sample being titrated. In an acid-base titration analysis, there should be a sudden change in pH when the reaction is complete. For example, if the sample being titrated is an acid, then the titrant to be used will be basic. When one excess drop of titrant is added, the solution being titrated will suddenly become basic. There are various natural and synthetic dyes, called indicators, which exist in different colored forms at different pH values. A suitable indicator can be chosen that will change color at a pH value consistent with the point at which the titration reaction is complete. The indicator to be used in this experiment is phenolphthalein, which is colorless in acidic solutions, but changes to a pink form at basic pH.

APPARATUS

Burette with stand
Pipette
1-L volumetric flask with stopper
250 mL Erlenmeyer flasks
Retort stand with clamp

As conclusion, the result that we get for part B,

As conclusion, average molarity of NaOH solution (M) in part B is 0.0900 M.
For part C, the average molarity of

REAGENTS

Sodium hydroxide pellets
Potassium hydrogen phthalate (KHP)
Phenolphthalein
Unknown vinegar

PROCEDURE

Part A: Preparation of the Sodium Hydroxide Solution

1. Clean and rinse a 1-L volumetric flask and stopper. Label the flask "Approx. 0.1 M NaOH". Put about 500 mL of distilled water into the flask.
2. Weigh out approximately 4.0 g of sodium hydroxide pellets and transfer to the 1-L flask. Stopper and shake the flask to dissolve the sodium hydroxide.
3. When all the sodium hydroxide pellets have dissolved, add additional distilled water to the bottle until the mark on the neck of the flask. Stopper and shake thoroughly to mix.

Part B: Standardization of the Sodium Hydroxide Solution

1. Set up the burette in the burette clamp. Rinse and fill the burette with the freshly prepared sodium hydroxide solution.
2. Clean three 250-mL Erlenmeyer flasks with water, and then rinse with distilled water. Label them as 1, 2, and 3
3. Remove the bottle of dried KHP from the oven. When the KHP is completely cool, weigh three samples of KHP between 0.6 and 0.8 g, one for each of the Erlenmeyer flasks. Record the exact weight of each KHP sample to the nearest mg (±0.001 g).
4. Add 100 mL of distilled water to KHP sample 1. Add 2 – 3 drops of phenolphthalein indicator solution. Swirl to dissolve the KHP sample completely.
5. Record the initial reading of the NaOH solution in the burette to the nearest 0.02 mL.
6. Add NaOH solution from the burette to the sample in the Erlenmeyer flask, swirling the flask constantly during the addition.
7. When the titration is approaching the endpoint, add NaOH one drop at a time, with constant swirling, until one single drop of NaOH causes a permanent pale pink color that does not fade on swirling. Record the reading of the burette to the nearest 0.02 mL.
8. Repeat step 4 – 7 with the other 2 KHP samples.
9. Given that the molecular mass of KHP is 204.2, calculate the number of moles of KHP in samples 1, 2, and 3.
10. From the number of moles of KHP present in each sample, and from the volume of NaOH solution used to titrate the sample, calculate the molar concentration (M) of NaOH in the titrant solution. The reaction between NaOH and KHP is of 1 : 1 stoichiometry.

Part C: Analysis of a Vinegar Solution

Vinegar is a dilute solution of acetic acid and can be effectively titrated with NaOH using the phenolphthalein endpoint.

1. Clean three Erlenmeyer flasks, and label as samples 1, 2, and 3.
2. Rinse the 5-mL pipette with small portions of the vinegar solution and discard the rinsings.
3. Using the pipetter, pipette 5 mL of the vinegar solution into each of the Erlenmeyer flasks. Add about 100 mL of distilled water and 2 – 3 drops of phenolphthalein indicator solution to each flask.
4. Refill the burette with the NaOH solution and record the initial reading of the burette to the nearest 0.02 mL. Titrate Sample 1 of vinegar in the same manner as in the standardization until one drop of NaOH causes the appearance of the pink color.
5. Record the final reading of the burette to the nearest 0.02 mL.
6. Repeat the titration for the other two vinegar samples.
7. Based on the volume of vinegar sample taken, and on the volume and average concentration of NaOH titrant used, calculate the molar concentration of the vinegar solution.
8. Given that the formula mass of acetic acid is 60.0, and the density of the vinegar solution is 1.01 g/mL, calculate the percent by mass of acetic acid in the vinegar solution.

QUESTIONS

1. Give the definition of indicators.

2. Suppose a NaOH solution were to be standardized against pure solid primary standard grade KHP. If 0.4538 g of KHP requires 44.12 mL of the NaOH to reach a phenolphthalein endpoint, what is the molarity of the NaOH solution?

3. Commercial vinegar is generally $5.0 \pm 0.5\%$ acetic acid by weight. Assuming this to be the true value for your sample, by how much were you in error in your analysis?

Experiment 1: Acid and Base Titrations

Data Sheet

Name:

Student No.:

Date:

Part A: Standardization of the Sodium Hydroxide Solution

Particulars	Trial 1	Trial 2	Trial 3
Mass of KHP taken (g)	0.705	0.710	0.700
Final burette reading (mL)	40.3	39.0	38.0
Initial burette reading (mL)	2.1	0.6	0.0
Volume of NaOH used (mL)	38.2	39.6	38.0
Molarity of NaOH solution			
Average molarity of NaOH solution			

Part B: Analysis of a Vinegar Solution

Particulars	Trial 1	Trial 2	Trial 3
Volume of vinegar solution used (mL)	5	5	5
Final burette reading (mL)	40.6	39.8	
Initial burette reading (mL)	0	0.1	0.4
Volume of NaOH used (mL)			
Molarity of NaOH solution			
Molarity of vinegar solution			
% mass of acetic acid in vinegar			
Average molarity of vinegar solution			
Average % mass of acetic acid in vinegar			

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EXPERIMENT 2: WATER ANALYSIS: SUSPENDED SOLIDS AND DISSOLVED OXYGEN

INTRODUCTION

Water is a very important resource for human being. A large portion of our body is water. A healthy human being can live for weeks without foods, but will not survive for days without water. When you are thirsty and need a drink of water, do you take for granted that the tap water is safe to drink? Do you think about what might be dissolved or suspended in the water?

Water in the environment has a large number of impurities. Dissolved solids are water-soluble substances, usually salts. Naturally occurring dissolved solids generally result from the movement of water over mineral deposits, such as limestone. These dissolved solids generally consist of the sodium, calcium, magnesium, and potassium cations and the chloride, sulfate, bicarbonate, carbonate, bromide, and fluoride anions. The dissolved solids are responsible for the "hard" water that exists in some locales. Anthropogenic (human-related) dissolved solids include nitrates from fertilizer runoff and human wastes, phosphates from detergents and fertilizers, and organic compounds from pesticides and industrial wastes. Salinity, a measure of the dissolved solids in a water sample, is defined as the grams of dissolved solids per kilogram of water.

Suspended solids are very finely divided particles that are water insoluble but are filterable. These particles are kept in suspension by the turbulent action of the moving water. Examples of suspended solids include decayed organic matter, sand, salt, and clay. Total solids are the sum of the dissolved and suspended solids in the water sample. In this experiment the total solids and the dissolved solids are determined directly; the suspended solids are assumed to be the difference, since ***total solids = dissolved solids + suspended solids***

The dissolved oxygen is determined based on the iodine/thiosulfate method. Excess potassium iodide is added to the acidified water sample, the dissolved oxygen (or other oxidizing agents that are present) will oxidize the iodide ions to iodine (in the form of triiodide, I_3^-). The iodine formed will then be determined by titration with the standard solution of sodium thiosulfate with starch solution as the indicator.

APPARATUS

Burette with stand
Pipette
Beaker
250 mL Erlenmeyer flasks
Evaporation dishes
Watch glass
Filter funnel
Filter paper

REAGENTS

KI
0.05M $Na_2S_2O_3$ solution
Starch solution
6M Sulfuric acid

PROCEDURE:

I: Determination of Suspended Solids in a Water Sample

1. Clean, dry, and determine the mass (to the nearest milligram, ±0.001 g) of two evaporation dishes. Be certain that you can identify each. Use the same balance for the remainder of the experiment.

Part IA: Determination of dissolved solids

1. Gravity filter about 50 mL of a thoroughly stirred or shaken water sample into a clean, dry 100 mL beaker. While waiting for the filtration to be completed, proceed to Part B.
2. Pipette a 25 mL portion of the filtrate into one of the evaporating dishes. Determine the mass of the combined evaporating dish and sample. Place the dish containing the sample on the wire gauze and heat slowly (do not boil) the mixture to dryness. As the mixture nears dryness, cover with a watch glass, and reduce the intensity of the flame. If spattering does occur, allow the dish to cool to room temperature, rinse the adhered solids from the watch glass and return the rinse to the dish.
3. Again heat slowly, and avoid further spattering. After all of the water has evaporated, maintain a small flame beneath the dish for 3 minutes. Allow the dish to cool to room temperature and determine its final mass.

Part IB: Determination of total solids and suspended solids

1. Thoroughly agitate 100 mL of sample; pipette a 25 mL aliquot of this sample into the second evaporating dish. Evaporate the sample to dryness as described in Part A.
2. Calculate the mass of total and suspended solids in the original water sample, using data from Part A.

Part IC: Analysis of data

1. Compare your data with three other groups in your laboratory who analyzed the same water sample. Record their results on the data sheet.
2. Calculate the standard deviation of the suspended solids from the four analyses on the water sample.

II: Determination of Dissolved Oxygen in a Water Sample

1. Prepare three clean Erlenmeyer flasks.
2. Clean a burette with tap water, then rinse with distilled water, and finally rinse with the 0.05M sodium thiosulfate standard solution. Then fill the burette with the sodium thiosulfate solution and record its initial volume to the nearest 0.02 mL

3. Using a clean pipette, transfer 25 mL of the water sample into each of the Erlenmeyer flasks and label as sample 1, 2 and 3.
4. Add about 2 g of KI and 10 mL of 6M sulfuric acid to sample 1. Iodide ions will be oxidized to iodine, and then it is titrated with the standard sodium thiosulfate solution.
5. On titration, the color of sample will become lighter. Stop adding the $Na_2S_2O_3$ solution when the color of the sample becomes light yellow that means it has nearly reached the end point. Now add 2 – 3 mL of starch solution to the sample. The sample will appear as dark-blue color.
6. Continue adding the $Na_2S_2O_3$ solution drop by drop until the dark-blue color just disappears. Record the final volume to the nearest 0.02 mL
7. Repeat the titration with the sample 2 and 3. From the concentration and the volume of the $Na_2S_2O_3$ solution used in the titration, calculate the concentration of oxygen present in the water sample.

QUESTIONS

1. In evaporating a solution to dryness in an evaporating dish, why must the heating rate be decreased as the mixture nears dryness?

2. How do pollutants such as sewage lower the dissolved oxygen content of water sources?

3. A 25 mL aliquot of a well-shaken sample of river water is pipetted into 27.211 g evaporating dish. After the mixture is heated to dryness, the dish and remaining sample has a mass of 43.617 g. Determine the total solids in the sample, express in units of g/kg sample. Assume the density of the sample to be 1.01 g/mL.

Experiment 2: Water Analysis: Suspended Solids and Dissolved Oxygen

Data Sheet

Name:

Student No.:

Date:

I: Determination of Suspended Solids in a Water Sample

Part IA: Determination of dissolved solids

Particular	Data
Mass of evaporating dish (g)	40.483
Mass of evaporating dish + water sample (g)	64.786
Mass of water sample (g)	24.303
Mass of evaporating dish + dried sample (g)	
Mass of dissolved solids in 25 mL aliquot (g)	
Mass of dissolved solids per total mass of sample (g solids/g sample)	
Dissolved solids (g solids/kg sample)	

Part IB: Determination of total solids and suspended solids

Particular	Data
Mass of evaporating dish (g)	41.037
Mass of evaporating dish + water sample (g)	65.860
Mass of water sample (g)	24.823
Mass of evaporating dish + dried sample (g)	41.000
Mass of total solids in 25 mL aliquot (g)	0.037
Mass of total solids per total mass of sample (g solids/g sample)	
Suspended solids (g solids/kg sample)	

Part IC: Analysis of data

Particular	Group 1	Group 2	Group 3	Group 4
Dissolved solids (g/kg)				
Total solids (g/kg)				
Suspended solids (g/kg)				
Average Suspended solids (g/kg)				

II: Determination of Dissolved Oxygen in a Water Sample

Concentration of $Na_2S_2O_3$ standard solution: _____

Particular	Trial 1	Trial 2	Trial 3
Final burette reading (mL)	0.9	1.5	3.3
Initial burette reading (mL)	0	0.4	2.3
Volume of $S_2O_3^{2-}$ used (mL)	0.9	1.1	1.0
Moles of $S_2O_3^{2-}$ used (mol)			
Volume of water sample (mL)			
Concentration of sample (M)			
Mean concentration of sample (M)			

0.6 — 3.3

0.2

2.6 — 2.8

2.9 — 3.4

22.7 23.2 22.7

25 25 25

5.

22

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EXPERIMENT 3: WATER ANALYSIS: HARDNESS OF WATER AND CHLORIDE ION/CHLORINE ION CONCENTRATION

INTRODUCTION

What does it mean when we say that water is "hard"? Hard water contains the dissolved salts of calcium, magnesium, and iron ions which are called hardening ions. In low concentrations these ions are not considered harmful for domestic use, but at higher concentrations these ions interfere with the cleansing action of soaps by forming insoluble compounds with soaps. Soaps, sodium salts of fatty acids such as sodium stearate, $C_{17}H_{35}CO_2Na$, are very effective cleansing agents so long as they remain soluble; the presence of the hardening ions however causes the formation of a grey, insoluble soap scum such as $(C_{17}H_{35}CO_2)_2Ca$. This grey precipitate appears as a "bathtub ring" and it also clings to clothes, causing white clothes to appear grey.

In industries, hard water can accelerate the corrosion of steel pipes, especially those carrying hot water. It is also responsible for the formation of "boiler scale" on tea kettles and pots used for heating water. The boiler scale is a poor conductor of heat and thus reduces the efficiency of transferring heat. Boiler scale also builds on the inside of hot water pipes to decrease the flow of water; in extreme cases, this buildup causes the pipe to break. Boiler scale consists primarily of the carbonate salts of the hardening ions. Ground water becomes hard as it flows through underground limestone ($CaCO_3$) deposits; Surface water similarly accumulates hardening ions as a result of it flowing over limestone deposits. Because of the relative large natural abundance of limestone deposits and other calcium minerals, it is not surprising that Ca^{2+} ion, in conjunction with Mg^{2+}, is a major component of the dissolved solids in water.

Hardness Classification of Water

Hardness (ppm $CaCO_3$)	Classification
< 15 ppm	Very soft water
15 ppm – 50 ppm	Soft water
50 ppm – 100 ppm	Medium hard water
100 ppm – 200ppm	Hard water
> 200 ppm	Very hard water

The concentration of the hardening ions in a water sample is commonly expressed as though the hardness is due exclusively to $CaCO_3$. The unit for hardness is mg $CaCO_3$/L, which is also ppm $CaCO_3$.

In this experiment a titration technique is used to measure the combined Ca^{2+} and Mg^{2+} concentration in a water sample. The titrant is EDTA, the disodium salt of ethylenediaminetetraacetic acid (abbreviated Na_2H_2Y).

In aqueous solution Na_2H_2Y dissociates into Na^+ and H_2Y^{2-} ions. The H_2Y^{2-} ion reacts with the hardening ions, Ca^{2+} and Mg^{2+}, to form very stable complex ions, especially in a solution buffered at a pH of about 10. An ammonia-ammonium ion buffer is often used for this pH adjustment in the analysis. A special indicator called Eriochrome Black T (EBT) is

used to detect the endpoint in the titration. EBT forms complex ions with Ca^{2+} and Mg^{2+} ions, but binds more strongly to Mg^{2+} ions. Because only a small amount of EBT is added, only a small quantity of Mg^{2+} is complexed; no Ca^{2+} ion is complexed to EBT, therefore most of the hardening ions remain "free" in solution. The EBT indicator is blue in solution but the $[Mg\text{-}EBT]^{2+}$ complex ion is red.

$$\underset{}{Mg^{2+}(aq)} + \underset{\text{blue}}{EBT(aq)} \longrightarrow \underset{\text{red}}{[Mg\text{-}EBT]^{2+}(aq)}$$

Before any H_2Y^{2-} titrant is added for the analysis, the solution is red. As H_2Y^{2-} titrant is added, it complexes with the "free" Ca^{2+} and Mg^{2+}.

$$Ca^{2+}(aq) + H_2Y^{2-}(aq) \longrightarrow CaY^{2-}(aq) + 2H^{+}(aq)$$

$$Mg^{2+}(aq) + H_2Y^{2-}(aq) \longrightarrow MgY^{2-}(aq) + 2H^{+}(aq)$$

Once the H_2Y^{2-} form complexes with all of the "free" Ca^{2+} and Mg^{2+} from the water sample, it will remove the Mg^{2+} from the $[Mg\text{-}EBT]^{2+}$ complex; the solution turns from red back to blue color, and the endpoint is reached.

$$\underset{\text{red}}{[Mg\text{-}EBT]^{2+}(aq)} + H_2Y^{2-}(aq) \longrightarrow MgY^{2-}(aq) + 2H^{+}(aq) + \underset{\text{blue}}{EBT(aq)}$$

For the endpoint to appear Mg^{2+} must be present; therefore a small amount of MgY^{2-} is usually added to the buffer solution. The added Mg^{2+} does not affect the amount of H_2Y^{2-} used in the analysis because an equimolar amount of Na_2H_2Y is also added.

The concentration of chloride ions in water sample can be determined by Mohr precipitation titration. The water sample is titrated with standard silver nitrate solution and sodium chromate is used as an indicator. A small amount of calcium carbonate is added to change the pH of the water sample to basic condition.

APPARATUS

Volumetric flask
Burette with stand
Pipette
Beaker
250 mL Erlenmeyer flasks

REAGENTS

Na_2EDTA
Standard Ca^{2+} solution
Mg/EDTA solution
Ammonia-ammonium ion buffer solution
Eriochrome Black T
0.05M $AgNO_3$ solution
Sodium chromate, Na_2CrO_4
Calcium carbonate, $CaCO_3$

PROCEDURE

I: Determination of the hardness of a water sample

Part IA: To prepare a standard 0.01M Na_2EDTA solution

1. Measure about 1.25 g (±0.01 g) of Na_2EDTA (molar mass = 372.24 g/mol); transfer it to a 250 mL volumetric flask containing about 200 mL of distilled water and stir to dissolve. Dilute to the "mark" on the volumetric flask with distilled water.
2. Prepare a burette for titration. Rinse a burette with the Na_2EDTA solution and then fill it with the Na_2EDTA solution. Record the initial volume (±0.02 mL) of the solution.
3. Pipette out 25.0 mL of the standard Ca^{2+} solution provided into a 250 mL Erlenmeyer flask, and record its molar concentration. Then add 2 mL of the buffer (pH = 10) solution, 5 mL of Mg/EDTA solution, and 5 – 6 drops of EBT indicator. Titrate the standard Ca^{2+} solution with the Na_2EDTA solution; swirl continuously. Near the endpoint, slow the rate of addition to drops; the last few drops should be added at 3 – 5 seconds intervals. The solution changes from red to purple to blue; the solution is blue at the endpoint.
4. Repeat the titration twice, then calculate the concentration of the Na_2EDTA solution.

Part IB: Analysis of the hardness of water sample

1. Obtain about 100 mL of a water sample. If the water sample is too turbid, you will need to gravity filter the sample before the analysis, and if the sample is acidic, add 1M NH_3 until it is basic to litmus.
2. Pipette out 25 mL of the water sample into a 250 mL Erlenmeyer flask, add 2 mL of the buffer (pH = 10) solution, 5 mL of Mg/EDTA solution, and 5 – 6 drops of EBT indicator, then titrate with the Na_2EDTA solution till the endpoint is reached. Repeat the titration thrice and then determine the hardness of the water sample.

II: Determination of the concentration of chloride ion

1. Rinse a clean burette with some standard 0.05M $AgNO_3$ solution, and then fill it with the $AgNO_3$ solution. Record the initial volume (±0.02 mL) of the solution.
2. Pipette 25 mL of the water sample into an Erlenmeyer flask. Add 3 – 5 drops of sodium chromate solution (yellow color) and a piece of pea size calcium carbonate.
3. Titrate the water sample slowly with the standard 0.05M $AgNO_3$ solution from the burette with constant swirling. At the beginning, a white precipitate, AgCl is formed. As soon as all the Cl^- ions have been precipitated, a red precipitate, Ag_2CrO_4, starts to form. Stop the titration when the endpoint is reached, that is when the red color remains.
4. Repeat the titration twice and then determine the concentration of the chloride ions present in the water sample.

QUESTIONS

1. Why was it necessary to add a small amount of magnesium/EDTA complex to the calcium sample before titration?

2. Chloride determination of a 25 mL well water sample requires 34.32 mL of 0.05012 M $AgNO_3$ solution to reach a sodium chromate endpoint. Calculate the concentration of chloride ion in the water sample.

Experiment 3: Water Analysis: Hardness Of Water And Chloride Ion/Chlorine Ion Concentration

Data Sheet

Name:

Student No.:

Date:

I: Determination of the hardness of a water sample

Part IA: To prepare a standard 0.01M Na_2EDTA solution

Molarity of Ca^{2+} solution: _____

Particulars	Trial 1	Trial 2	Trial 3
Volume of standard Ca^{2+} solution used (mL)			
Final burette reading (mL)			
Initial burette reading (mL)	0.00		
Volume of Na_2EDTA used (mL)			
Molarity of Na_2EDTA solution (M)			
Average molarity of Na_2EDTA solution (M)			

Part IB: Analysis of the hardness of water sample

Particular	Trial 1	Trial 2	Trial 3
Final burette reading (mL)			
Initial burette reading (mL)			
Volume of Na_2EDTA used (mL)			
Moles of Na_2EDTA used (mol)			
Volume of water sample (mL)			
Concentration of sample (M)			
Mean concentration of sample (M)			

II: Determination of the concentration of chloride ion/chlorine ion

Particular	Trial 1	Trial 2	Trial 3
Final burette reading (mL)			
Initial burette reading (mL)			
Volume of $AgNO_3$ used (mL)			
Moles of $AgNO_3$ used (mol)			
Volume of water sample (mL)			
Concentration of sample (M)			
Mean concentration of sample (M)			